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**Advanced Photorefractive Materials Processing
for Holographic Data Storage**

Status Report for the Month of November

Thermal fixing of holograms in photorefractive media requires doping by a suitable ionic carrier. In LiNbO_3 it is well-established that these carriers are protons, but in other materials that have also been shown to exhibit some degree of fixing, such as KNbO_3 and BaTiO_3 , the source of the carriers is still uncertain, although oxygen vacancies are a likely choice. In order to pursue thermal fixing, we have now begun experiments into hydrogen loading of LiNbO_3 and SBN. This work complements our on-going oxidation/reduction experiments as reported last month, with the future goal being the optimized combination of the two treatments, either by using (1) a single optimized high-temperature treatment, or (2) a distinct two-step method in which the material is first optimally reduced, followed by a second low-temperature high-pressure hydrogen loading treatment which is designed to minimize any change in the oxidation/reduction state.

Several LiNbO_3 samples (with dimensions $10 \times 10 \times 2 \text{ mm}^3$) were prepared for a variety of experiments. Four samples were used for in-house hydrogen doping studies. Our treatments were based on high-temperature annealing (4 hours at 950°C) in dry and wet O_2 atmospheres. The wet processing was conducted by bubbling O_2 through water which was held at 20 and 70°C . The experiments were designed to determine the degree of control that we can expect to have over the proton concentration, $[\text{H}^+]$. Quantitative determinations of $[\text{H}^+]$ were made by measuring the OH^- absorption coefficient α_{oh} at $2.87 \mu\text{m}$ and using the most recent determination of the absorption cross section, $\sigma_{\text{oh}} = 9.4 \times 10^{-20} \text{ cm}^2$, as reported by Muller *et al.* [Appl. Phys. Lett. 60, 3212 (1992)]. The results, based on $[\text{H}^+] = \alpha_{\text{oh}}/\sigma_{\text{oh}}$, are displayed in Fig. 1, where the proton concentration is expressed in terms of a mol% doping level. The first important point to note here is that the proton concentration can easily be made to approach 1 mol%, which is comparable to the heaviest Fe doping levels used for photorefractive purposes. This means that the available dynamic range for recording fixed holograms should not be limited by the proton concentration, but instead by the efficiency with which the photorefractive space-charge distribution is transferred to the corresponding proton distribution. The second point to note is that the proton concentration is, in fact, readily controllable, with our measured

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values spanning a range from $5.3 \times 10^{18} \text{ cm}^{-3}$ to $1.32 \times 10^{20} \text{ cm}^{-3}$. Similar experiments are in progress with SBN.

An additional eight LiNbO_3 samples have also been sent to AT&T Bell Labs for the high-pressure hydrogen loading experiments. Before shipping, these samples were annealed in a dry O_2 atmosphere to remove residual hydrogen. Transmission spectra were also taken so that the change in hydrogen concentration as well as any change in the visible absorption can be precisely measured following the AT&T treatments. Two separate high-pressure experiments will be conducted using temperatures of 20°C and 275°C . In both cases the nominal hydrogen pressure will be maintained near 350 atm.

To achieve maximum photorefractive dynamic range (i.e., maximum space-charge fields), it is desirable to have an $\text{Fe}^{2+}:\text{Fe}^{3+}$ concentration ratio approximately equal to one. For $\text{Fe}:\text{LiNbO}_3$ annealed in dry argon-oxygen mixtures, the equilibrium Fe oxidation state is completely determined by the temperature and oxygen partial pressure. Based on the work of Phillips and Staebler [J. Electron. Mat. 3, 601 (1974)], we estimate the optimal partial pressure to be quite low, about 1.5 torr (0.002 atm) for annealing temperatures of 900 to 1050°C . The addition of water vapor to the annealing gas may have a strong influence on the optimal oxygen pressure due to the liberation of oxygen as protons enter the LiNbO_3 lattice. This effect, however, should be transitory, lasting only until the equilibrium proton concentration is established in the crystal. We are currently modifying our crystal processing system in order to conduct high-temperature treatments with a very carefully controlled oxygen partial pressure.

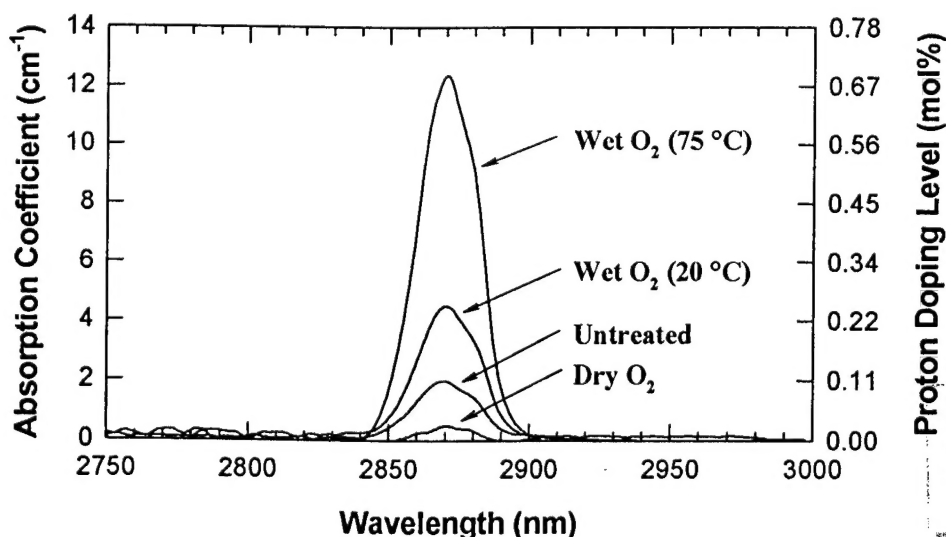


Figure 1. Absorption spectra taken across the OH^- absorption line in LiNbO_3 for various processing treatments, illustrating a large degree of control over the proton doping concentration.

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